

# **VERIFICATION OF TRANSLATION**

I, Roy Nishimura, translator of 3F Karasumanijyo Bldg., 267,
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hereby declare that I am conversant with the English and Japanese languages
and am a competent translator thereof. I further declare that to the best of
my knowledge and belief the following is a true and correct translation made
by me of Japanese Patent Application number 2002-338627 filed on
November 21, 2002.

Date: September, 1, 2006

Roy Nishimura



[Name of the Document]

Request for Patent

[Docket Number]

165-095

[Filing Date]

November 21, 2002

[Destination of Filing]

Commissioner of the Patent Office, Shinichiro Oda

[International Patent Classification]

G01N 15/02

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[Indication of Filing Fee]

[Register Number]

192752

[Amount of Payment]

21,000 yens

[List of Communications]

[Name of Object]

Specification

1

1

[Name of Object]

Drawings

[Name of Object] Abstract 1

[Necessity of Proof] Necessary

[Name of the Document]

Specification

[Title of the Invention]

Particle Size Distribution Analyzer

[Claims]

[Claim 1] A particle size distribution analyzer comprising: a transparent cell for containing a sample containing particles to be analyzed; a laser light irradiating section for irradiating the sample with laser light from outside of the cell; a scattering light intensity detecting section for detecting the intensity of light scattered from the particles irradiated with laser light; a calculating section for calculating a particle size distribution of the particles based on a fluctuation of the intensity of scattering light measured which occurs due to Brownian motions of the particles; and a noise reducing section operative to reduce the amount of noise causing scattering light becoming incident on the scattering light intensity detecting section, the noise reducing section comprising a region to be irradiated with laser light of at least one of outside surface and inside surface of the cell, the region being inclined a predetermined angle with respect to the optical axis of laser light.

[Claim 2] The particle size distribution analyzer in accordance with claim 1, wherein the scattering light intensity detecting section is configured to measure the intensity of back scattering light which travels in reverse of a direction of irradiation of laser light on the sample.

[Detailed Description of the Invention]

[0001]

[Technical Field to which the Invention Pertains]

The present invention relates to dynamic light scattering particle size distribution analyzers.

[0002]

[Conventional Technique]

Conventionally, the so-called "dynamic light scattering particle size distribution analyzer", as disclosed in Patent Document 1, is known as a device for measuring the particle diameters of small particles. This type of device analyzes a particle size distribution based on the following measuring principle. That is, in a sample containing a certain concentration of particles to be measured, the particles move irregularly (Brownian motion) in a solvent with the tendency that relatively large particles move slowly while relatively small particles move quickly. When the sample is irradiated with laser light having a fixed frequency, the laser light is scattered with the particles to be measured. Since the particles are individually in Brownian motion as described above, the frequency of scattering light from each particle shifts slightly from the frequency of incident light depending on the motion velocity of each particle due to Doppler shift.

[0003]

Therefore, measurement of such shifts in frequency makes it possible to determine the particle size distribution of the sample statistically. Specifically, the total sum of frequency shifts from all the particles is observed as a "fluctuation" in the intensity of scattering light and, hence, the particle size distribution can be calculated from such a fluctuation by using an algorithm exclusive to this type of calculation.

[Patent Document 1] Patent Laid-Open Publication No. 2002-221479

[Problems to be solved by the Invention]

However, such scattering light takes place not only at the particles to be measured but also at scratches of a cell containing the aforementioned sample, the interface between the cell and outside air, the interface between the cell and the sample, and the like. Such scratches and interfaces are fluctuated due to 1/f fluctuation occurring in nature, minute fluctuations of the cell, presence of surface tension wave, or

the like. Scattering light from such scratches and interfaces are also subject to frequency shifts due to such fluctuations and hence cause noise to occur, which affects the measurement.

### [0005]

On the contrary, with a sample having a relatively high concentration, since scattering light from particles to be measured are predominant, signals generated by noise causing scattering light scattered from the aforementioned scratches and the like are of negligible levels because they are buried in optical fluctuation signals from the particles. However, in the case of a sample having a relatively low concentration of particles to be measured or a sample containing fine particles, signals generated by noise causing scattering light is of levels that cannot be neglected and as a whole suffers from such a problem as to lower the measurement precision or the SN ratio, which might result in impossibility of effective measurement.

# [0006]

With a view to overcoming this problem, a dynamic light scattering particle size distribution analyzer has heretofore been developed of the immersed cell type in which a cell is immersed in a liquid having a refractive index approximately equal to that of the cell in order to reduce noise-causing scattering light occurring at the interface between the outside surface of the cell and outside air due to a difference in refractive index.

#### [0007]

Such an analyzer, however, cannot effectively eliminate noise-causing scattering light caused by scratch or deflection of the cell wall and noise-causing scattering light occurring at the interface between the inside surface of the cell and the sample contained therein, though it is capable of eliminating noise-causing scattering light occurring at the interface between the outside surface of the cell and the outside.

Particularly recently, heavy use has been made of a disposable cell made of a resin such as a plastic. Such a cell is susceptible to scratch, deflection, or streak resulting from heterogeneity of a plastic and appearing like a string in the cell wall, which are responsible for the occurrence of noise-causing scattering light.

## [0008]

Further, the immersed cell type analyzer often uses an organic solvent to immerse the cell. Such an organic solvent is difficult to handle and often requires that the structure of the analyzer be made complicate. In addition, dust or contaminant, which is possibly mixed into the solvent, may cause noise causing scattering light to occur.

## [0009]

Accordingly, it is an object of the present invention to provide a dynamic light scattering particle size distribution analyzer having a very simple structure capable of reducing noise-causing scattering light that is responsible for the occurrence of noise.

#### [0010]

# [Means to solve the problems]

In order to accomplish the aforementioned object, the present invention provides a particle size distribution analyzer includes a transparent cell for containing a sample containing particles to be measured, a laser light irradiation section for irradiating the sample with laser light from outside of the cell, a scattering light intensity detecting section for detecting the intensity of scattering light from the particles irradiated with laser light, and a calculating section for calculating a particle size distribution of the particles based on a fluctuation of the intensity of scattering light measured which occurs due to Brownian motions of the particles, where at least a region to be irradiated with laser light at outside—surface or/and inside surface of the cell is inclined a predetermined angle with respect to the optical axis of laser light in order to reduce an

amount of noise-causing scattering light, which is caused due to scratches on the cell, the difference in refractive index between the cell and outside air or the like, becoming incident on the scattering light intensity detecting section.

## [0011]

The term "inclined", as used herein, means a state where the wall surface forms an angle except 90 degrees with the optical axis of laser light.

### [0012]

Such a construction can employ a very simple arrangement such that an existing cell, for example, is mounted at a different angle than in a conventional analyzer, to realize a large reduction in the influence on measurement from noise causing scattering light which is caused by minute unevenness (scratch) on the cell surface or streaks in the cell wall or which occurs at the interface between the outside surface of the cell and outside air or at the interface between the inside surface of the cell and the sample due to a difference in refractive index between them. Since the intensity distribution direction of such noise causing scattering light greatly depends on the angle of irradiation of laser light on the outside surface or inside surface of the cell, inclining the outside surface or/and inside surface of the cell will make it possible to cause noise causing scattering light to travel in directions different from the direction of the optical axis of scattering light to be measured.

#### [0013]

Further, such an inclined cell wall surface is capable of reliably cutting the amount of light reflected by the cell wall surface, thereby largely reducing the noise component derived from reflected light.

## [0014]

Thus, the analyzer of the present invention can lower the noise level substantially and hence is capable of effectively analyzing even a sample having a very

low particle concentration which could not be analyzed in the past.

[0015]

In a specific embodiment of the present invention which makes the effect of the invention more noticeable, the scattering light intensity detecting section is configured to measure the intensity of back scattering light which travels in reverse of a direction of irradiation of laser light on the sample. The expression "back scattering light which travels in reverse of a direction of irradiation of laser light on the sample", as used herein, is meant to include light rays having directional components reverse to the direction of irradiation of laser light.

[0016]

[Modes for Carrying Out the Invention]

One embodiment of the present invention will be described with reference to the drawings.

A particle size distribution analyzer according to this embodiment, as shown in Figs. 1 and 2, is of the dynamic light scattering type and includes a transparent cell 2 for containing a sample containing particles C to be measured, a cell unit 3 holding the transparent cell 2 therein, a laser light irradiating section 4 for irradiating the sample with laser light having a certain frequency from outside of the cell 2, a scattering light intensity detecting section 5 for detecting the intensity of light scattering from the particles C irradiated with laser light and outputting the intensity detected as an intensity signal, an intensity signal receiving section 6 for receiving the intensity signal, and a calculating section 7 for calculating the particle size distribution of the particles C based on a fluctuation of the intensity signal received by the intensity signal receiving section 6.

[0017]

Description will be made of each part of the analyzer 1.

# [0018]

The cell 2 includes, for example, a hollow rectangular parallelepiped transparent glass member having a uniform thickness and is removably held by a non-illustrated cell holder provided in the cell unit 3. The sample contained in the cell 2 is a dispersion containing a relatively low concentration of particles C in a dispersing medium such as water. The particle concentration may be relatively high when the particles C are fine. Since Brownian motions of the particles C in the sample vary sensitively with change in temperature, which may affect the measurement, this embodiment is provided with a temperature control mechanism (not shown in the drawings) for controlling the temperature within the cell unit 3 to stabilize the temperature of the sample under measurement, thereby realizing high-precision measurement.

# [0019]

The laser light irradiating section 4 includes a light source of laser light having a semiconductor laser 41 for example, and a light guide mechanism 42 defining an optical axis for guiding laser light irradiated from the semiconductor laser 41 to the sample. The light guide mechanism 42 includes a collimator lens 43 for turning diffused laser light irradiated from the semiconductor laser 41 into a beam of parallel laser light having a certain diameter, and a condenser lens 44 for condensing the beam of parallel laser light onto a predetermined irradiated region set at a location slightly inwardly of the inside surface of the cell 2.

#### [0020]

The scattering light intensity detecting section 5 includes a detecting unit (photodetector) 51 for receiving light of a predetermined wave range and outputting an intensity signal which is an electric signal corresponding to the intensity of light received, and a scattering light guide mechanism 52 defining an optical axis for guiding

scattering light, which scattered from the particles C, to the photodetector 51.

# [0021]

The scattering light guide mechanism 52 functions to cause scattering light, which is scattered in a direction opposite to the traveling direction of incident laser light by particles C present in the aforementioned irradiated region, to travel in reverse to the traveling direction of incident laser light along the same optical axis up to some midpoint. The scattering light guide mechanism 52 includes a paralleling lens 53 for turning scattering light into a beam of parallel light having a larger diameter than the aforementioned beam of parallel laser light, a noise light cutoff portion 54 for cutting off noise light such as multiple scattering light from the beam of paralleled scattering light, and a second condenser lens 55 for condensing the scattering light outgoing from the noise light cutoff portion 54 onto the light-receiving surface of the photodetector 51. The paralleling lens 53 also serves as the aforementioned condenser lens 44, so that the optical axis of scattering light coincides with the optical axis of incident laser light up to some midpoint.

#### [0022]

The noise light cutoff portion 54 includes a shielding plate 541 having a pinhole PH, and a pair of convex lenses 542 and 543 positioned on opposite sides of the shielding plate 541. The noise light cutoff portion 54 functions to condense the beam of paralleled scattering light into the pinhole PH with one lens 542 and then again parallel the beam of scattering light re-diffusing after having passed through the pinhole PH with the other lens 543.

## [0023]

This embodiment further includes a reflecting mirror 56 between the noise light cutoff portion 54 and the second condenser lens 55 for reflecting scattering light outgoing from the noise light cutoff portion 54 so that it can be received by the light-

receiving surface of the photodetector 51 through the second condenser lens 55. Since the reflecting mirror 56 is disposed on the optical axis of the beam of parallel laser light, the reflecting mirror 56 centrally defines a laser light passing hole LH having a diameter generally equal to the diameter of the beam of parallel laser light so as to allow the beam to pass therethrough without varying the amount of parallel laser light.

## [0024]

The intensity signal receiving section 6 comprises a preamplifier 61 and an A/D converter 62 as basic components and functions to receive the intensity signal input as an analog signal and output a digital signal converted from the analog signal.

# [0025]

The calculating section 7 functions to calculate the particle size distribution of the particles C based on a fluctuation of the intensity signal output from the intensity signal receiving section 6 and then output the result of calculation in a predetermined form to a display or a printer through an information processing device 8 such as a personal computer. While this embodiment employs a homodyne detection method utilizing fluctuations resulting from interference between light scattered from the particles C, it is needless to say that the present invention may employ a heterodyne detection method. It should be noted that since the details of the calculating section 7 and the algorithm used in the calculating section 7 have been described in Japanese Patent Laid-Open Publication No. 2000-171383 and the like by the inventors of the present invention, description thereof is herein omitted.

## [0026]

In this embodiment, at least a region of the cell 2 which is to be irradiated with laser light and allows laser light to pass therethrough is inclined a certain angle with respect to the optical axis of the laser light. Specifically, the mounting angle of the whole cell 2 is set so that front wall 21 of the cell 2 on which laser light becomes

incident forms an angle of 45 degrees for example with the optical axis of the incident laser light.

## [0027]

This arrangement makes it possible to largely reduce (for example 1/100) noise-causing scattering light which is caused by fine unevenness (scratch) on the surface of the cell 2 or streaks in the cell wall or which occurs at the interface between the outside surface 21a of the cell 2 and outside air or between the inside surface 21b of the cell 2 and the sample due to the difference in refractive index between them, as compared to an arrangement where the front wall 21 of the cell is positioned normal to the optical axis of laser light, that is, the front wall 21 forms an angle of 90 degrees with the optical axis of laser light.

# [0028]

The intensity of such noise-causing scattering light greatly depends on the angle of irradiation of laser light on the outside surface 21a or inside surface 21b of the cell 2. In the aforementioned arrangement where the front wall 21 is positioned normal to the optical axis of laser light, laser light that has become incident on the front wall 21 scatters intensely in the direction just opposite to the direction of irradiation of laser light, thus causing a high level of noise. In contrast, the arrangement where the front wall 21 is inclined as shown in Figs. 1 and 2 causes the traveling direction of noise-causing scattering light including the angle of diffusion of the noise-causing scattering light to differ from the direction of irradiation of laser light, i.e. the direction of the optical axis of scattering light to be detected.

## [0029]

Further, this embodiment is capable of reliably cutting off light reflected by the cell wall surfaces 21a and 21b, hence, reducing the noise component derived from such reflecting light.

# [0030]

As described above, this embodiment is further provided with the noise light cutoff portion 54 and hence is capable of cutting off noise-causing scattering light that is scattered at locations other than the predetermined irradiated region located adjacent the inside surface 21b of the cell 2 into which laser light is to be condensed, as well as multiple scattered light. Accordingly, this embodiment can be expected to exhibit a noise reducing action based on synergy with the noise light cutoff portion 54.

### [0031]

As a result, this embodiment makes it possible to analyze the particle size distribution of even a sample having a very low particle concentration (for example 1/100 of a conventionally analyzable concentration) effectively.

# [0032]

It is to be noted that the present invention may be modified variously.

# [0033]

For example, though the existing cell is mounted at an angle different from the conventional mounting angle in this embodiment, it is possible to make the angle of irradiation of laser light different from the conventional angle of irradiation of laser light. Further, the cell may be configured into a triangular prism as shown in Fig. 3. The essential feature of the present invention is that at least a region to be irradiated with laser light of the outside surface and/or inside surface of the cell and the optical axis of laser light are inclined a predetermined angle relative to each other.

## [0034]

Of course, the wall of the cell need not necessarily have a uniform thickness and only one of the inside surface and outside surface of the cell may be inclined relative to the optical axis of irradiation of laser light. Such an arrangement can also produces a desired noise reducing effect. Further, the noise light cutoff portion is not necessarily

needed and hence may be eliminated in view of the principle of the invention.

[0035]

The present invention is not limited to the embodiment described above with the drawings and may be modified variously without departing from the spirit of the present invention.

[0036]

[Effect of the Invention]

According to the present invention having been described in detail, a very simple arrangement such that an existing cell, for example, is mounted at a different angle than in a conventional analyzer, makes it possible to largely reduce the influence on measurement by noise causing scattering light which is caused by minute unevenness (scratch) on the cell surface or streaks in the cell wall or which occurs at the interface between the outside surface of the cell and outside air or between the inside surface of the cell and the sample due to the difference in refractive index between them. Since the intensity distribution direction of such noise causing scattering light greatly depends on the angle of irradiation of laser light on the outside surface or inside surface of the cell, inclining the inside surface or inside surface of the cell will make it possible to cause noise light to travel in a direction different from the direction of the optical axis of scattering light to be measured.

Thus, the analyzer of the present invention can lower the noise level substantially and hence is capable of effectively analyzing the particle size distribution of even a sample having a very low particle concentration or containing fine particles.

[Brief Description of the Drawings]

[Fig. 1]

A schematic view illustrating the overall construction of a dynamic light scattering particle size distribution analyzer in accordance with one embodiment of the

present invention;

[Fig. 2]

An enlarged fragmentary sectional view of a relevant portion of the analyzer shown in Fig. 1

[Fig. 3]

A schematic view illustrating the overall construction of a dynamic light scattering particle size distribution analyzer in accordance with another embodiment of the present invention

[Explanation of Letters or Numerals]

1 · · · dynamic light scattering particle size distribution analyzer

 $2 \cdot \cdot \cdot \text{cell}$ 

21a · · · outside cell wall surface

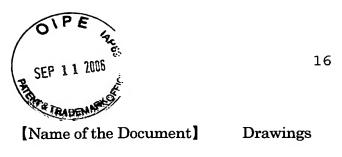
21b · · · inside cell wall surface

4 · · · laser light irradiating section

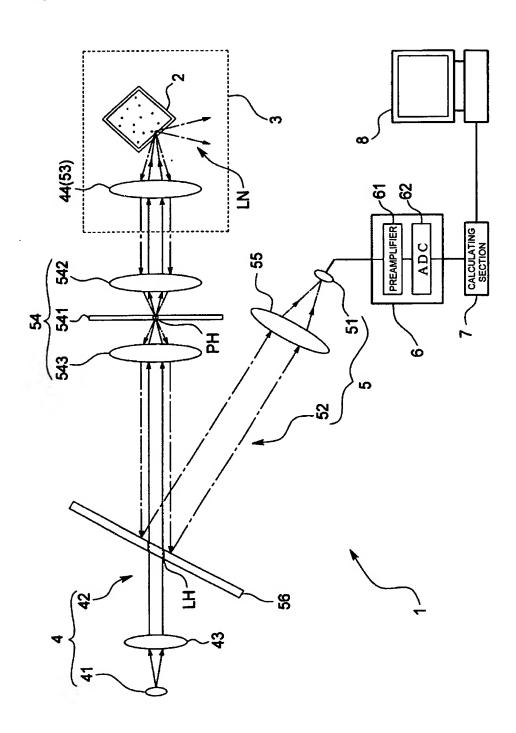
 $5 \cdot \cdot \cdot$  scattering light intensity detecting section

7 · · · calculating section

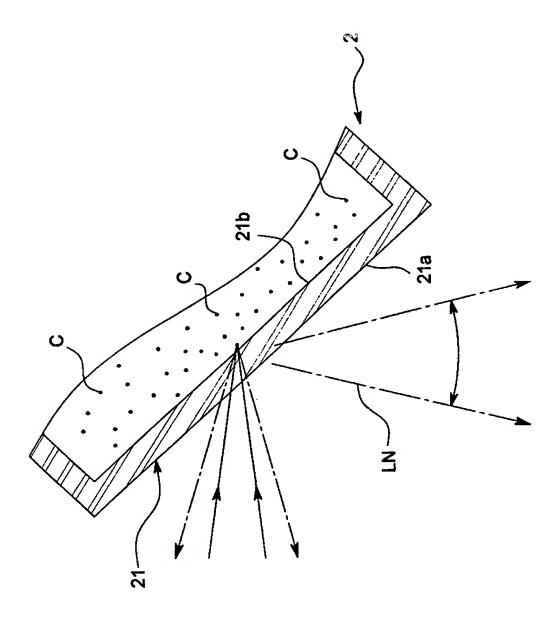
 $C \cdot \cdot \cdot$  particle to be analyzed



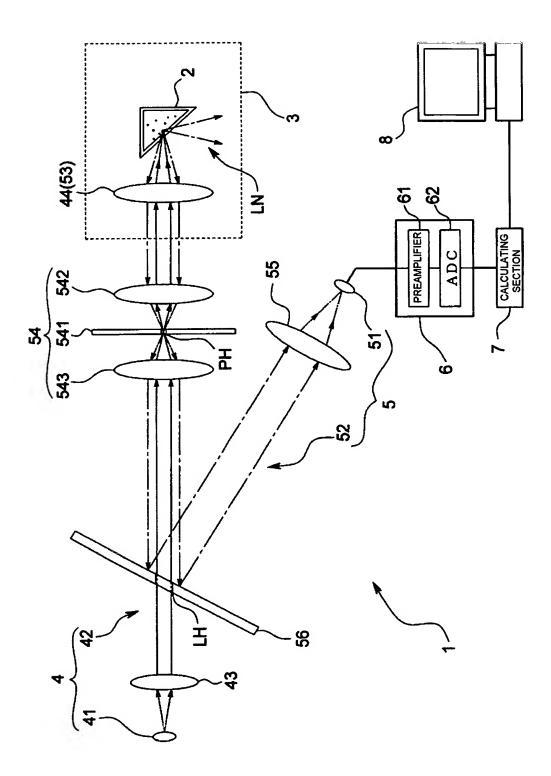
[Fig. 1]



[Fig. 2]



[Fig. 3]





[Name of the Document]

**Abstract** 

[Abstract]

[Problem] To provide a dynamic particle size distribution analyze with very simple structure capable of reducing noise-causing scattering light.

[Means to Solve the Problem] The particle size distribution analyzer includes: a transparent cell (2) for containing a sample containing particles (C) to be analyzed; a laser light irradiating section (4) for irradiating the sample with laser light from outside of the cell (2); a scattering light intensity detecting section (5) for detecting the irradiation of light scattered from the particles (C) irradiated with laser light; and a calculating section (7) for calculating a particle size distribution of the particles (C) based on a fluctuation of the irradiation of scattering light detected which occurs due to Brownian motions of the particles (c), where at least a region irradiated with laser light at an outside cell surface (21a) or/and an inside cell surface (21b) of the cell is inclined at a predetermined angle relative to an optical axis of laser light in order to reduce an amount of the noise causing scattering light, which is caused due to scratches on the cell (2), the difference in a refractive index between the cell (2) and outside air or the like, becoming incident on the scattering light intensity detecting section.

[Selected Drawing] Fig. 1